Electrochemical Nanoindentation Study on Influence of Hydrogen on Local Mechanical Properties of Fcc Metals at Slow Strain Rate

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A nanoindentation apparatus with an electrochemical cell for cathodic hydrogen charging and a wide dynamic range of load duration time was developed, and influence of hydrogen on local mechanical properties of Type 316L stainless steel, pure Ni, and three kinds of Ni–Cr bialloy (56Ni–43Cr, 69Ni–30Cr, 79Ni–20Cr) was investigated. For Type 316L stainless steel and pure Ni, nanohardness was increased by about 10% and unchanged, respectively, by hydrogen charging at load duration time between 0.1 s and 10 800 s. On the other hand, for Ni–Cr bialloys, degree of nanohardness increase by hydrogen charging was reduced with increase in load duration time. As a result, for 69Ni–30Cr and 79Ni–20Cr, hydrogen caused softening at long load duration time of more than 600 s. Hydrogen embrittlement susceptibility of each sample was also evaluated by slow-strain rate tensile tests, and compared with the observed nanoindentation results. The tendency was found that, for samples with higher hydrogen embrittlement susceptibility, softening is caused by hydrogen charging at long load duration time.

KEY WORDS: nanoindentation; hydrogen embrittlement; stainless steel; nickel; Ni–Cr bialloy.

1. Introduction

Hydrogen embrittlement (HE) is a phenomenon where hydrogen degrades strength or toughness of high-strength steel, and remains a major problem in steel industry. Although HE has been extensively investigated for decades, there are still many uncertainties on its mechanism. One of difficulties in the HE problem arises from the complexity that hydrogen is trapped at various kinds of microstructure such as a grain boundary, a precipitate, and a micro void, and interaction between hydrogen and the individual microstructure is not clear.¹⁾ Meanwhile, information on influence of hydrogen on mechanical properties such as hardness and plasticity is important because HE is a kind of fracture phenomenon. Therefore, in order to elucidate the HE mechanism, characterization of influence of hydrogen on mechanical properties of small volume by means of local analytical tools is expected. Historically, nanoindentation (NI) has been employed to measure hardness, elastic modulus, and elastic limit of thin-film materials^{2,3)} or miniature structures,^{4,5)} and is considered promising for this purpose.

At room temperature, hydrogen diffuses in metals and finally desorbs from the metal surface. This means that, even if hydrogen is fully charged into a sample, hydrogen concentration at the subsurface region immediately drops in the air.⁶⁾ On the other hand, NI is known to be a surface sensitive method, and a typical indentation depth is less than 1 μm . Thus, for the application of NI to the HE problem,

in-situ measurements in hydrogen are required. The first *in-situ* measurements have been conducted by A. Barnoush *et al.* with an electrochemical nanoindentation (EC-NI), where indentation was performed in electrolyte during cathodic hydrogen charging.^{7–9)} In load-displacement curves of NI measurements, a discrete jump of the displacement with respect to the load, referred to as a “pop-in”, is often observed at elasticity limit. A. Barnoush *et al.* have found that pop-in load is lowered during hydrogen charging for many metals (*e.g.*, Al, Cu, Ni,⁷⁾ FeAl,⁸⁾ and Fe-3%Si⁹⁾), and proposed that hydrogen lowers critical shear stress for homogeneous dislocation nucleation.

However, it is expected that strain rates widely used in the previous NI studies are too fast to cause HE. It is well known that HE becomes pronounced at slower strain rates, where solute hydrogen atoms can follow mobile dislocations. For instance, in slow strain-rate tensile tests (SSRT) of pure iron, hydrogen changes neither fracture elongation nor morphology of fracture surface at strain rates of more than $10^{-4}/\text{s}$.¹⁰⁾ Equivalent strain ε around an imprint formed by a spherical indenter with a curvature radius of R is given by the following equation:

$$\varepsilon \cong \frac{1}{5} \frac{\sqrt{R^2 - (R - h_c)^2}}{R}, \quad \dots\dots\dots (1)$$

where h_c is a depth of the imprint.¹¹⁾ Substituting typical numerical values ($R=1 \mu\text{m}$, and $h_c=200 \text{ nm}$) into Eq. (1), $\varepsilon \approx 0.1$ is obtained. Load duration time Δt of NI apparatus is typically a few seconds, and therefore, a strain rate ($\cong \varepsilon/\Delta t$) is estimated to be about $10^{-2}/\text{s}$, which is much faster than

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the strain rates where HE appears in SSRT.

Technically, Δt is limited to below a few minutes because thermal drift (TD) of the indenter and the sample gradually changes the indenter-sample distance by time. Thus, in the present study, an EC-NI apparatus with a wide dynamic range of Δt was developed by compensating TD. Moreover, influence of hydrogen on mechanical properties of fcc metals was investigated by the developed EC-NI apparatus, and the observed results were compared with HE susceptibility evaluated by SSRT.

2. Experimental

2.1. Method of Thermal Drift Compensation

In NI measurements, relation between applied load P and indenter displacement h as illustrated in **Fig. 1** is recorded under load control. The recorded P - h curve is analyzed by an Oliver and Pharr (O-P) method to obtain nanohardness and elastic modulus (Young's modulus).¹²⁾ In this method, a gradient S , referred to as a "contact stiffness", of the unloading segment of the P - h curve at a maximum load $P_{\max}$ is obtained by a curve-fitting technique. Then, h_c is calculated from the relation,

$$h_c \cong h_{\max} - 0.75 \frac{P_{\max}}{S}, \dots\dots\dots (2)$$

where $h_{\max}$ is a maximum displacement. It is noted that $h_{\max}$ is not always equivalent to h_c because the sample is elasto-plastically deformed by the indenter. Elastic modulus E_r and nanohardness H_n are obtained by substituting $P_{\max}$, h_c and S into the Sneddon's equation,¹³⁾

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c(h_c)}}, \dots\dots\dots (3)$$

and the equation,

$$H_n = \frac{P_{\max}}{A_c(h_c)}. \dots\dots\dots (4)$$

The function $A_c(x)$ in Eqs. (3) and (4) is a projected area of an imprint with a depth of x , and its form is obtained from

Fig. 1. Schematic of a load-displacement curve obtained in nanoindentation measurements.

indentations into a standard specimen (e.g., fused silica). The distance between the sample and the indenter is gradually changed by time because of TD of the indenter and the sample. As a result, when Δt is long, the P - h curves are distorted, causing inaccuracy both in E_r and H_n .

In the present study, in order to compensate TD and to realize NI measurements at longer Δt , P - S curves were recorded. Additionally, E_r was separately measured by the O-P method at short Δt , where influence of TD is negligible. The change in S can be recorded in real time by modulating P and by detecting the synchronized signals in h with a lock-in amplifier.¹²⁾ By substituting the measured S and E_r into the Sneddon's equation in Eq. (3), h_c at given load P was obtained. Subsequently, P - h and P - H_n curves were calculated by substituting, S , P and the obtained h_c into Eqs. (2) and (4), respectively. In principle, S measured instantaneously with the lock-in technique is hardly affected by TD. Hence, the P - h and P - H_n curves calculated with the above procedures are also not affected by TD.

2.2. Procedure

Type 316L stainless steel (Type316L), pure Ni, and three kinds of Ni-Cr bialloy (56Ni-43Cr, 69Ni-30Cr, and 79Ni-20Cr) with chemical composition in **Table 1** were used as samples for the EC-NI measurements. In order to remove residual strain, commercial cold-rolled sheets of Type316L and pure Ni were annealed in an infrared vacuum furnace at 1 270 K for 1 800 s and 900 s, respectively, followed by furnace cooling. On the other hand, as-forged samples were used for Ni-Cr bialloys. The forging temperature was set to about 1 270 K. The sample surfaces, where an indenter is pressed, were mechanically polished with abrasive papers and alumina compounds, and finished by electrochemical polishing. It was identified with atomic force and optical microscopes that all the samples have equiaxed grains, and approximate grain size is 20 μm for Type316L, 200 μm for pure Ni, and commonly 50 μm for the Ni-Cr bialloys. It was also confirmed by X-ray diffraction and transmission electron microscopy (TEM) that all the Ni-Cr bialloys are fully composed of fcc phase and no bcc phase exists.

The EC-NI measurements were performed with a commercial nanoindentation apparatus (Hysitron TriboIndenter 950) with a custom-build electrochemical cell as illustrated in **Fig. 2**. Volume of electrolyte in the electrochemical cell is about 1 cm^3 . Hydrogen was charged into the sample at room temperature using borate buffer solution (pH. 8.6) and a platinum counter electrode under current control. Cathodic current density was commonly set to 2.5 A/m^2 for all the samples. For the indenter, a spherical diamond indenter

Table 1. Chemical composition of the fcc metals used in the present study. The unit is mass%.

	C	Si	Mn	Cr	Fe	N	Ni	Mo
56Ni-43Cr	0.009	0.03	0.5	43	0.14	0.0037	56.11	—
69Ni-30Cr	0.004	0.01	0.5	30.27	0.12	0.0031	69.17	—
79Ni-20Cr	0.002	<0.01	0.51	20.22	0.18	0.0027	79.41	—
pure Ni	—	—	—	—	—	—	99	—
Type316L	<0.03	<1	2	16–18	Bal.	—	10–14	2–3

Fig. 2. Schematic of the electrochemical nanoindentation apparatus developed in the present study.

Table 2. Chemical composition of the Ni–Cr bialloys used in SSRT.

	C	P	S	Cr	Ni	Annealing temperature [K]
60Ni-39Cr	0.002	<0.001	0.002	39.7	60.29	1 370
70Ni-29Cr	0.002	<0.001	0.002	29.68	70.31	1 320
80Ni-19Cr	0.002	<0.001	0.002	19.85	80.15	1 250

with $R=1\text{ }\mu\text{m}$ was used. In order to compensate TD, P was modulated at 200 Hz with Hystron's nanoDMA III option. The load amplitude was dynamically changed during indentation, and set to 1.25% or 3% of P . E_r used for the TD compensation was separately obtained from indentations at $\Delta t=5\text{ s}$. The indenter was pressed into grain interiors under load control, and influence of hydrogen was investigated for the same grain at the same Δt ; different grains were pressed at different Δt . P_{max} was chosen so that h_{max} becomes around 200 nm, and strain rate was controlled by changing Δt between 0.1 s and 10 800 s.

SSRT was performed to investigate HE susceptibility of each sample. For Ni–Cr bialloys, the samples with chemical composition in Table 2 was used, whereas, for the other fcc metals, the same samples as in Table 1 were used. For recrystallization, the Ni–Cr bialloys were annealed at temperatures given in Table 2, and a grain size of 100 μm was commonly obtained. Tensile test specimens with 2.54 mm diameter and 25.4 mm gage length without a notch were strained at a strain rate of $3\times 10^{-4}\text{ s}^{-1}$ at room temperature. For the electrolyte, 1N- H_2SO_4 was used, and 1.4 kg/m^3 of $\text{CH}_4\text{N}_2\text{S}$ was added as a poison. Before the tests, hydrogen was precharged for 10 800 s at cathodic current density of 300 A/m^2 . During the tests, on the other hand, cathodic current density was decreased to 10 A/m^2 . The specimens were strained until they are fractured. Morphology of the fracture surface was observed by scanning electron microscopy.

3. Results

3.1. EC-NI Results

3.1.1. Preliminary Experiment

First of all, it was examined if the TD compensation described in Section 2.1 is obtained using 56Ni-43Cr as a sample. P - h curves with and without the TD compensation

Fig. 3. Load-displacement curves of 56Ni-43Cr obtained (a, b) in the air and (c) in electrolyte without hydrogen charging at $\Delta t=3\text{ }600\text{ s}$. The curves in (a) were obtained without thermal drift compensation and those in (b) and (c) with thermal drift compensation.

at $\Delta t=3\text{ }600\text{ s}$ in the air are shown in Figs. 3(a) and 3(b), respectively. P - h curves with the TD compensation in electrolyte without hydrogen charging are also shown in Fig. 3(c). In order to examine reproducibility, three curves were obtained by pressing the indenter into the same grain in the same experimental conditions. Because S is not obtained with a lock-in technique during unloading, only the loading segments of the P - h curve were plotted in the figures. Some spikes were seen in the P - h curves with the TD compensa-

tion at small loads. Generally, A_c is inaccurate for very small h_c . As a result, h_c numerically obtained from Eq. (3) using E_r and S is erroneously diverged at small loads, which results in the observed spikes. As expected, a large variation was seen for the P - h curves without the TD compensation, whereas the variation was suppressed for the P - h curves with the TD compensation both in the air and in electrolyte. It was confirmed that the TD compensation is successfully applied at Δt up to 10 800 s.

3.1.2. Influence of Hydrogen

Influence of hydrogen on local mechanical properties at a fast strain rate ($\Delta t=5$ s) was investigated by comparing P - h curves obtained with and without hydrogen charging. Changes in H_n obtained by the O-P method are shown in Fig. 4(a). The value of H_n in the figures is an average of about 15 data, and the error bar is their standard deviation. During hydrogen charging, H_n was increased by about 30% for 56Ni-43Cr, and by about 10% for Type316L. In contrast, hydrogen charging did not affect H_n of pure Ni, 69Ni-43Cr, and 79Ni-20Cr. It was found that the degree of H_n increase observed for 56Ni-43Cr was different among different grains. However, large increase by more than 20% was always observed for this sample. When hydrogen charging was stopped, H_n returned to the original value before the hydrogen charging. The observed change in H_n is therefore caused by diffusive hydrogen charged into the samples. Changes in E_r and those in pop-in load by hydrogen charging are also shown in Figs. 4(b) and 4(c), respectively. For all the samples, hydrogen charging changed neither E_r nor pop-in load.

It has been reported in the EC-NI study by A. Barnoush *et al.* that pop-in load of pure Ni is decreased by hydrogen charging, which is different from the present results.⁷⁻⁹⁾ Pop-in load is known to be strongly affected by dislocation density, surface roughness, and also an apex condition of the indenter.¹⁴⁾ It seems that the discrepancy in the influence of hydrogen on the pop-in load arises from difference in these experimental conditions.

Next, Δt dependence of influence of hydrogen was investigated using TD compensation. P - h curves of 69Ni-30Cr at Δt of 5 s, 600 s, and 10 800 s are shown in Figs. 5(a) to 5(c), respectively. The P - h curves at $\Delta t \leq 600$ s are an average of five curves, and those at $\Delta t > 600$ s are an average of three curves. At $\Delta t=5$ s, traces of the P - h curves with and without hydrogen charging were identical, indicating that H_n was not changed by hydrogen charging. On the other hand, at longer Δt , a maximum displacement of the P - h curves was increased during hydrogen charging, indicating that H_n was decreased.

In Fig. 6, the relation between Δt and the change in H_n corresponding to the data in Fig. 5 is plotted. In the figure, results of the other samples were also given. For different Δt , H_n of different grains was measured. Therefore, in order to eliminate crystalline orientation dependence, the change in H_n was represented by the rate ΔH_n calculated from the following equation:

$$\Delta H_n = \left(\frac{H_n^{\text{withH}}}{H_n^{\text{w/oH}}} - 1 \right) \times 100, \dots\dots\dots (5)$$

Fig. 4. Influence of hydrogen charging on (a) nanohardness, (b) elastic modulus, and (c) pop-in load at $\Delta t = 5$ s.

where H_n^{withH} and $H_n^{\text{w/oH}}$ are H_n with and without hydrogen charging at the same P , respectively. Error bars of ΔH_n were shown for 56Ni-43Cr as a representative. A degree of error was almost the same for the other samples. For Ni-Cr bialloys, ΔH_n was decreased with increase in Δt . As a result, for 69Ni-30Cr and 79Ni-20Cr, hydrogen-induced softening was seen at $\Delta t \geq 600$ s. On the other hand, for Type316L and pure Ni, ΔH_n were about 10% and around zero, respectively, irrespective of Δt .

3.2. Slow Strain-rate Tensile Test

Reduction in fracture elongation by hydrogen charging and morphology of fracture surface of the hydrogen-charged specimen are summarized in Table 3. For Type316L, fracture elongation was not significantly reduced by hydrogen

Fig. 5. Load-displacement curves of 79Ni-20Cr with and without hydrogen charging at Δt of (a) 5 s, (b) 600 s, and (c) 10 800 s.

charging, and a dimple fracture surface was observed. For pure Ni, fracture elongation was reduced by about 20% by hydrogen charging, and a dimple surface and a quasi-cleavage one were observed for the center and the periphery of the fracture surface, respectively. On the other hand, for Ni-Cr bialloys, degree of fracture elongation reduction by hydrogen charging was increased with increase in Ni content, and an intergranular fracture occurred; the reduction in fracture elongation was about 30% for 60Ni-39Cr, 70% for 70Ni-29Cr, and 90% for 80Ni-19Cr. Thus, it was identified that the sample with the lowest HE susceptibility used in the present study is Type316L, followed by pure Ni, 56Ni-43Cr, 69Ni-30Cr, and 79Ni-20Cr.

Fig. 6. Relation between Δt and ΔH_n . Error bars are shown for 56Ni-43Cr as a representative.

Table 3. Reduction in fracture elongation by hydrogen charging, and morphology of fracture surface in SSRT.

	Reduction in fracture elongation by hydrogen charging (%)	Morphology of fracture surface
60Ni-39Cr	25	Intergranular
70Ni-29Cr	66	Intergranular
80Ni-19Cr	89	Intergranular
pure Ni	18	Quasi-cleavage & dimple
Type316L	3	Dimple

4. Discussion

It is recognized from the ΔH_n - Δt relation in Fig. 6 that hydrogen charging tends to increase H_n at fast strain rates, and to decrease H_n at slow strain rates. It is also recognized from comparison between the ΔH_n - Δt relation in Fig. 6 and the HE susceptibility in Table 3 that hydrogen charging causes softening at slow strain rates for samples with higher HE susceptibility. In Fig. 7, relation between ΔH_n at $\Delta t = 3\,600$ s and reduction in fracture elongation in SSRT is plotted to clarify this relationship. Because the data in Fig. 6 are somewhat dispersed and have a large error, ΔH_n at $\Delta t = 3\,600$ s was determined by fitting the data in Fig. 6 to a straight line in logarithmic scale,

$$\Delta H_n = A \log \Delta t + B, \dots\dots\dots (6)$$

where A and B are fitting parameters. It can be confirmed from Fig. 7 that larger softening is caused for the samples with higher HE susceptibility such as 79Ni-20Cr, whereas softening is not caused for the samples with lower HE susceptibility such as Type316L.

The precise mechanism of the observed tendency that hydrogen causes hardening at fast strain rates and softening at slow strain rates is still not clear. However, it is likely that change in dislocation mobility by hydrogen may contribute. S. Taketomi *et al.* have investigated competitive motion between hydrogen and an edge dislocation in α -Fe

Fig. 7. Relation between reduction in fracture elongation by hydrogen charging in SSRT and ΔH_n at $\Delta t = 3600$ s.

by atomic simulations, and found that hydrogen increases dislocation mobility at slow strain rates and decreases at fast strain rates.¹⁵⁾ The increase and decrease in dislocation mobility result in hardening and softening, respectively. Although the detailed calculations are necessary, the same phenomena as those in α -Fe are likely to occur also in fcc metals. According to the calculations by S. Taketomi *et al.*, the degree of softening (or hardening) is dominated by hydrogen diffusion coefficient, energy barrier for dislocation motion, or hydrogen concentration. It is considered that the observed difference in ΔH_n - Δt relation among the different samples in Fig. 6 arises from the difference in these material properties.

Regarding the mechanism of the observed softening at slow strain rates, decrease in degree of work-hardening by hydrogen is also considered. H. K. Birnbaum *et al.* have investigated hydrogen-dislocation interaction by calculation and environmental TEM observation, and suggested that hydrogen weakens repulsive interaction among the dislocations because hydrogen shields elastic fields.^{16,17)} Meanwhile, K. Miyata has conducted TEM observations for Ni-Cr bialloys, which exhibited hydrogen-induced softening at slow strain rates in the present study, and reported that hydrogen enhances a cross-slip and increases screw components of dislocations.¹⁸⁾ When the cross-slip frequently occurs like this, dislocations can move around sessile dislocations, and work-hardening is suppressed. At slow strain rates, the hydrogen shield effect and the hydrogen-enhanced cross-slip become pronounced because there is enough duration time for hydrogen to accumulate around dislocations. Thus, it seems that the observed softening at slow strain rates is also attributed to decrease in degree of work-hardening due to the hydrogen shield effect or the hydrogen-enhanced cross-slip.

H_n measured in the present study are of grain interiors, whereas samples with higher HE susceptibility exhibited an intergranular fracture as shown in Table 3. In order to fully understand the correlation between HE susceptibility and degree of softening at slow strain rates in Fig. 7, fur-

ther information on interaction among dislocations, grain boundaries, and hydrogen, are necessary. For instance, TEM observations or mechanical tests of a microstructure containing a single grain boundary (e.g., micropillars¹⁹⁾ and microbending beams^{20,21)} would be useful for this purpose.

5. Summary

A nanoindentation apparatus with an electrochemical cell for cathodic hydrogen charging and a wide dynamic range of Δt was developed, and influence of hydrogen on local mechanical properties of five kinds of fcc metals (Type316L, pure Ni, 56Ni-43Cr, 69Ni-30Cr, and 79Ni-20Cr) was investigated. Furthermore, these observed results were compared with HE susceptibility evaluated by SSRT. As a result, the following findings were obtained.

1. For Type316L and pure Ni, H_n was increased by about 10% and unchanged irrespective of Δt . On the other hand, for Ni-Cr bialloys, degree of H_n increase by hydrogen charging was reduced with increase in Δt . Consequently, for 69Ni-30Cr and 79Ni-20Cr, hydrogen charging caused softening at $\Delta t > 600$ s.

2. There is the tendency that hydrogen charging increases H_n at fast strain rates, and decreases at slow strain rates. There is also the tendency that, for samples with higher HE susceptibility, softening is caused by hydrogen charging at slow strain rates.

3. The hydrogen-induced hardening at fast strain rates and the hydrogen-induced softening at slow strain rates are qualitatively explained by change in dislocation mobility or decrease in degree of work-hardening.

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